

SHORT COMMUNICATION

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On the structure of 9-amino-2,7-di-*tert*-butylacridine. By RICHARD E. MARSH, *The Beckman Institute, California Institute of Technology, Pasadena, California 91125, USA**

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Abstract

The crystal structure of 9-amino-2,7-di-*tert*-butylacridine hydrochloride monohydrate, $C_{21}H_{27}N_2^+.Cl^-.H_2O$, has been described in space group $P3_1$ [trigonal, $a = 11.086$ (2), $c = 14.367$ (4) Å, $Z = 3$; McKenna, Agbandje & Neidle (1992). *Acta Cryst.* **C48**, 188–190]. It is properly described in space group $P3_121$. Further refinement in $P3_121$, based on the original F values, leads to an improved R (0.049 vs 0.067) and to a revised position for the Cl^- ion.

A listing of 2795 F_{obs} values (apparently those with $F_{obs} \geq 6\sigma_F$) was obtained from Supplementary Publication No. SUP 54551 and averaged according to Laue symmetry $\bar{3}ml$, yielding a total of 767 independent reflections. This averaging showed a goodness of fit (GOF), with weights on an arbitrary but uniform scale, of 9.4 for 704 forms with multiple measurements and an R_{merge} of 0.029 for 157 forms represented by exactly two measurements. For averaging in point group $\bar{3}$, the corresponding GOF was 10.3 for 1069 multiple comparisons and R_{merge} was 0.038 for 600 pairs. In other words, averaging in the higher symmetry seemed to be an improvement, and leaves little doubt that $\bar{3}ml$ is to be preferred over $\bar{3}$.

Starting coordinates were from Table 1 of McKenna, Agbandje & Neidle (1992), symmetrized and averaged in accord with $P3_121$; necessary shifts were approximately as expected from the e.s.d.'s. A surprise was the position assigned to the Cl^- ion: it was constrained to lie 'on the crystallographic threefold axis', with $x = y = 0$. This is not

a special position in $P3_1$, and no such constraint is appropriate. In fact, Cl^- lies on a twofold axis in $P3_121$ with $y = 0$ (and $z = \frac{2}{3}$), but x is free to move.

Full-matrix least-squares minimization of $\sum w(F_{obs}^2 - F_{cal}^2)^2$, with weights w assigned according to Hughes (1941), converged at $R = 0.049$ for 117 parameters [including an extinction coefficient; final value $1.53(13) \times 10^{-6}$]. H atoms were placed in idealized positions and not refined. Computing was under the *CRYM* system (Duchamp, 1964), with scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV, pp. 99, 149); in view of the Laue symmetry assumed in averaging the data, the imaginary terms f'' were ignored. In the final cycle no parameter shifted by as much as 0.1 e.s.d. Final heavy-atom coordinates are given in Table 1;† with one exception, they are statistically indistinguishable from the starting $P3_1$ values. The exception is the Cl^- ion, whose y coordinate of 0.0195 (2) is over 0.2 Å from the 3_1 axis where it started. As a result, the hydrogen-bond lengths are changed, $N9 \cdots Cl^-$ from 3.380 (4) (average) to 3.300 (6) Å and $OW \cdots Cl^-$ from 3.034 (5) to 3.121 (6) Å.

One further comment seems appropriate. The water molecule *OW* and the *tert*-butyl groups show large, anisotropic displacement coefficients, suggesting the possibility of disorder. Indeed, the water molecule can be satisfactorily represented by two half-populated sites, with reasonable isotropic coefficients, about 0.8 Å apart and related by the added twofold symmetry axis. (A similar treatment of the *tert*-butyl groups was precluded by the more complicated patterns of displacement coefficients.) Thus, the twofold axis may only be a statistical one, possibly resulting from twinning. If this is the case the twinning is probably quite intimate, since there is no indication of deviation from Laue symmetry $\bar{3}ml$.

*Contribution No. 8595.

Table 1. *Coordinates, space group $P3_121$*

x, y, z and $U_{eq} \times 10^4$; $U_{eq} = (1/3)\sum_i \sum_j U_{ij} a_i^* a_j^* a_i \cdot a_j$.

	x	y	z	U_{eq}
C1	6012 (5)	−1094 (6)	4918 (3)	382 (13)
C2	5095 (6)	−1655 (6)	5638 (3)	365 (13)
C3	3835 (6)	−1644 (6)	5570 (3)	453 (15)
C4	3502 (6)	−1102 (6)	4824 (4)	449 (15)
C9	6654 (7)	0	3333	338 (19)
N9	7861 (6)	0	3333	453 (17)
N10	4130 (5)	0	3333	419 (16)
C11	5720 (5)	−531 (5)	4130 (3)	327 (13)
C12	4462 (5)	−532 (5)	4084 (3)	355 (13)
C15	5377 (6)	−2286 (7)	6494 (3)	529 (15)
C16	6636 (9)	−2432 (10)	6395 (6)	1159 (27)
C17	4170 (9)	−3717 (11)	6686 (7)	1541 (40)
C18	5505 (17)	−1448 (14)	7334 (5)	2295 (58)
Cl	195 (2)	0	8333	516 (5)
OW	1700 (6)	0	3333	1286 (29)

† Lists of structure factors, U_{ij} values and assumed H-atom coordinates have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 55437 (7 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: L11010]

References

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